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RADIATION AND THERMALLY HARDENED SWITCHING MATERIALS

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Over the past six months we have concentrated on the analysis of longevity factors of our devices, tried solutions to the difficulties we have recognized which are: Oxidation of the NbO_2 layer around the electrode contact and short, sputtering away of the W whisker's tip and wandering which makes for non-reproducibility of device characteristics. Potting of the assembly and use of an intermediate In dot seem to alleviate the problems. Further testing is in progress at Fort Monmouth. In addition we have developed new device structures:

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a) For the regular devices using cheaper Nb metal sheets instead of NbO single crystal chips.

b) For long intense pulses using directly single crystal NbO_2.

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INTRODUCTION

Over the past six months we have concentrated on the analysis of longevity factors of our devices, tried solutions to the difficulties we have recognized, and developed two new device structures expanding the domain of applications of the original discovery.

Among the factors which seem to influence the longevity of our devices, we have found that the main deterioration mechanism is the oxidation of the NbO_2 film at the contact point. This oxidation depends strongly on the polarity of the pulse. We have also found that the damage is lesser for compact, dense NbO_2 films than it is for the highly dendritic films obtained by fast oxidation. In this relation, we have been able to show that Titanium doping of the NbO substrate improves both the threshhold switching voltage and the resistance to damage of the NbO_2 film. This appears to be because Ti^{4+} cannot be further oxidized and therefore the average oxygen affinity for the doped surface has become lesser. Another improvement was to grow the NbO_2 film at lower temperature so that the kinetic is slowed down and a less dendritic, more dense film structure is obtained.

The other aspect of our work has been to develop a device structure such that it would be resistant to shock and present stabler, more reproducible switching properties. Electron micrographs have shown that the tungsten whiskers used for pressure contact tend to abrade and wander over devices surface. This of course makes for non-reproducibility of switching characteristics. We have attempted to correct this problem by potting the whole assembly and by using In at the end of the whisker to prevent abrasion of the tip. This approach has proven to be reasonably successful

with currents up to 40 Amps.

Finally, we have attempted to expand the domain of application of our devices in two directions:

1) We have tried to use Niobium metal sheet instead of NbO single crystals as substrates. This turned out to work successfully and would certainly be a cheaper approach to device construction.

2) In addition, for longer, more powerful pulses we have been able to develop a single crystal NbO_2 device. This type of device would clearly be more expensive but would also be unique inasmuch as it can handle millisecond pulses.

ANALYSIS OF LONGEVITY FACTORS

A) Oxidation

In order to investigate the deterioration mechanism in our devices, we have prepared chips of NbO with only one face covered with an NbO_2 thin film. We then applied the pulse via the tungsten whisker pressure contact to the NbO_2 thin film. This configuration resulted in a relatively fast deterioration of the film only when the polarity of the pulse was positive. In other words, the area around the point contact behaved as a cathode and pumped in oxygen from the atmosphere. If the polarity was reversed and the pulse was negative, the damage was considerably slower. The best configuration however consisted in applying the pressure contact to the NbO side. We assumed that such a structure would have a relatively high capacitance. To our surprise, we measured 1 pF instead of the usual 0.2 pF. We still cannot understand how it is that the capacitance in these devices does not seem to scale with the contact area. In any case, we found that when the pulse is applied to the NbO side, whether it is positive or

negative, the damage was considerably slower. We have been able to apply to these devices more than 10 million pulses without destroying them. Clearly when we talk about pulsing we always refer to our Velonex system with a maximum amplitude of the pulse of the order of 670 volts, rise times of 15 nanoseconds and a pulse width of 300 nanoseconds.

B) Film Quality

Numerous scanning electron microscope photographs made at Ft. Monmouth have shown that the NbO_2 film formed on our NbO single crystal chips is dendritic in nature, in other words needle-like crystallites grow up from the NbO surface. The average width of the whiskers is of the order of 1 to $1\frac{1}{2}$ micron. They are indicative of a very fast growth preventing the formation of large single crystal grains. This is clearly a disadvantage because the tungsten whiskers, or for that matter, whichever contact we choose to apply, will make contact only with a few grains and if it is maintained rigidly in place will short when these grains have become oxidized by repeated pulsing.

Another difficulty revealed by the scanning electron micrograph is that the tungsten whiskers" tip sputters away after a few pulses, and the end rounds off. This is no problem is spring action is maintained, however if somehow we rigidify the structure the contact is lost and the device opens.

With reference to the NbO_2 film structure we have attempted to palliate the difficulty in two ways. The first has been to grow the films at a lower temperature, namely 750°C instead of the usual 850°C. This makes for slower kinetics and better grain growth. Electron micrographs have borne out our reasoning and shown that films prepared in this way,

while still polycrystalline, are definitely denser to the point where grains actually cannot be resolved and the layer appears essentially 100% dense. A further improvement of the quality of the NbO_2 layer was obtained via a 10% Titanium doping of the NbO single crystal chips. These chips of course when oxidized produce a surface film of Titanium doped NbO_2. Given that Ti^{4+} cannot further oxidize, the doping in effect reduces the oxygen's affinity for the NbO_2 surface and we obtain a film less sensitive to the atmosphere, and therefore longer lived under repeated pulsing.

IMPROVEMENT OF DEVICE STRUCTURE

Our main effort has been to generate devices which performance is reasonably reproducible, in particular we concentrated on stability to shock and stability of electrode contact. These determine the I vs V characteristic as well as the off resistance.

A) Potting

The electron micrographs have shown that tungsten whiskers tend to wander all over the NbO_2 layer surface and therefore given a different contact situation over just about every pulse, it appeared reasonable to freeze in the situation by potting the whole assembly. While these new structures showed to be very stable, they were also very short lived. This was because the tungsten tip sputtered away and the contact happened since the spring action had been lost because of the potting.

B) Contact

This led us to our next step which was to put at the end of the tungsten whiskers a dot of In metal. The reason for this was that while

In melts at a relatively low temperature, it keeps a very low vapor pressure and therefore would not easily sputter away. The pulses, if they should melt the In would simply produce a better contact. This idea appears to work, at least for currents up to 40 Amps. In order to further confirm the validity of this approach, testing is ongoing at Ft. Monmouth by Lt. Laplante where currents up to 100 Amps will be applied. Only then will we know if these new assemblies are really successful.

NEW APPROACHES TO PREPARATION OF SWITCHING ELEMENTS

One of the aspects of the industrial production of our devices will necessarily be the growth of NbO single crystal chips. While this material grows relatively easily, it is definitely an expensive step. For this reason, it appeared reasonable to investigate the possibility of using Niobium metal sheets as substrates. We now know enough to do so. In effect we have established that the switching element in NbO_2, that the switching mechanism necessitates an NbO/NbO_2 junction, and that there is no intermediate oxide between NbO and NbO_2. Since Niobium metal exposed to an oxidizing atmosphere would first form NbO, then NbO_2, we would necessarily get the needed NbO/NbO_2 junction on top of an Nb electrode which should be no problem. The only question would be whether the general grain structure of the successive layer would turn out to be too fragile for repeated pulsing. We have prepared devices by simply exposing pieces of Niobium metal sheets to an oxidizing atmosphere produced by Nb_2O_5 powder at a temperature of $800^{\circ}C$ in evacuated ampulas. The resulting layers proved to be entirely satisfactory. In effect, the off

resistance was typically of the order of 30 kΩ and the threshold voltage in our system in the order of 140 volts. Longevity tests have shown that the devices are somewhat more fragile than those produced from NbO single crystal chips. They were able, however, to withstand at least 10,000 pulses. Clearly this is not yet at a stage where we could transcribe the procedure in industrial production terms. More work is needed if we want to follow this route.

From a different point of view, we have attempted to develop devices which would be slightly slower but would handle longer, more powerful pulses. The technique consisted in reducing the surface of NbO_2 single crystal platelets sliced off large NbO_2 single crystal boules. This reduction was effected by annealing in the presence of Niobium metal in evacuated ampulas at 1000°C. As we have already reported, it is possible to actually reduce NbO_2 very far without having it lose its crystallographic structure. What happens however is that the reduced layer acquires a lower resistivity. In effect, it becomes almost metallic. By this procedure we develop electrodes on each side of the platelets. It is then quite easy to bind silver wires, gold wires or to evaporate metallic counter electrodes to which such wires can be bound. In any case, we obtain device structures which are quite stable, present very reproducible I vs. V characteristics and can handle more intense pulses.

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